



***N-Methylpyrrolidone
Storage and Handling***

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Introduction

N-Methylpyrrolidone (NMP) is a highly polar, aprotic, general purpose organic solvent. A colorless, low-viscosity liquid with a faint amine odor, NMP is miscible in all proportions with water and conventional organic solvents.

As a solvent, NMP is employed in a wide range of industrial applications, including those listed here:

Process Chemicals

- Acetylene Recovery
- Butadiene Extraction
- Extraction of Aromatics
- Lube Oil Extraction
- Gas Purification

Engineering Plastics

- Spinning of Aramid Fibers
- Production of Polyphenylene Sulfide, PPS
- Preparation of Membranes for Ultrafiltration

Coatings

- Solvent for Ink Systems
- Component in Specialty and Waterborne Paints or Finishes
- Synthesis/Diluent of Wire Enamels
- Solvent for Plastic Coatings
- Coalescing Agent

Agricultural Chemicals

- Solvent and/or Cosolvent for Insecticides, Fungicides, Herbicides and Bioregulators
- Reaction Medium in Synthesis of Active Compounds

Electronic

- Cleaner for Silicon Wafers
- Photoresist Stripper in the Manufacture of Microchips
- Auxiliary in Printed Circuit Board Manufacturing

Paint Stripping and Cleaning

- Component in Paint Strippers, Degreasers and Industrial Cleaners
- Removal of Polyurethane Residues and Mold Release Agents
- Removal of Tarry or Polymeric Deposits from Reactors

Miscellaneous

- Solvent for Developer (Photo-Reproduction Technique)
- Manufacture of High-Performance Adhesives and Industrial Adhesive Removers
- Solvent for Slimicides Used in the Manufacture of Paper and Paperboard
- Dispersing Agent for Pigments
- Reaction Medium in the Synthesis of Organic Intermediates and Pharmaceuticals

This brochure presents basic information on appropriate precautions for the safe handling and storage of NMP. Additional information on physical properties, applications and toxicology is available from BASF Corporation.

Handling Precautions

Fire

NMP is considered a combustible material in bulk containers (larger than 55 gallons) by the U.S. Department of Transportation (DOT) because it has a flash point of 195.8°F (91°C). When ignited, NMP will sustain a fire. Handling facilities and equipment should be designed to minimize the probability of fire and should be equipped with proper firefighting equipment. All types of fire extinguishers are effective against NMP fires. However, the dry chemical extinguisher is recommended based on ease of operation. Firefighters should be equipped with self-contained breathing apparatus and turn-out gear.

Water makes an effective fire extinguishing material for NMP since it is miscible in water, unlike many other organic solvents. Alcohol-based foam is also recommended. Early review of these precautions with the appropriate local firefighting organization is recommended for all bulk storage locations.

Bulk storage tanks should be marked with the National Fire Protection Association (NFPA) fire hazard diamond symbol as set forth in National Fire Code Standard 704. The recommended hazard values for NMP given in the National Fire Code Guide 325M are: Fire – 2; Health (under fire conditions) – 2; Reactivity – 0.

Safety Precautions

Applicable codes which cover the storage and handling of NMP include:

1. OSHA 1910.106 (Occupational Safety and Health Administration)
2. NFPA No. 30 (National Fire Protection Association)
3. NEC Code Article 500 (National Electrical Code)

In addition to the above, all applicable federal, state and local ordinances, the requirements of underwriters and insurance companies, and the rules of good safety practices should be followed carefully.

Personal Protection and First Aid

Because NMP is an eye irritant, chemical splash goggles should be worn when handling it. If NMP enters the eye, flush with water for at least 15 minutes, and consult a physician.

Gloves of butyl rubber and FEP Teflon® provide the best resistance to NMP. Following use, gloves should be rinsed and discarded. Butyl rubber aprons may be used for splash protection; however, the PVC coatings found on most protective clothing dissolve rapidly in NMP.

If skin contact does occur, the affected area should be thoroughly flushed with water to prevent irritation, followed by moisturizing with a protective ointment.

If NMP is swallowed, dilute with plenty of water, induce vomiting immediately, and consult a physician.

Overexposure to NMP vapors can bring about nausea, headache or dizziness. Move the person to fresh air, aid in breathing, if needed, and consult a physician.

Sample Storage

NMP samples are stored safely in clear glass bottles or steel cans with sufficient vapor space to allow for thermal expansion. Bottles should be stored in a temperature-controlled environment. Polyethylene cap liners will prevent the breakdown of coated caps or liners. Electronic grade NMP samples should be stored in high density polyethylene bottles to avoid leaching of elemental components from glass.

Waste Disposal

NMP is classified as a combustible liquid. Waste NMP should be stored in approved safety-type disposal cans that are properly labeled as to their contents and hazard. NMP is highly biodegradable and may be treated effectively in an industrial wastewater treatment facility of the proper size using activated sludge technology. Similarly, regional municipal wastewater plants can often handle the normal daily discharge from NMP processing facilities. Nevertheless, disposal of NMP by these routes should not commence until approval has been obtained from treatment facility operators.

Operational procedures should be designed to avoid NMP spills and leaks. Should they occur, contain spills for subsequent recovery or disposal. Polyethylene or steel containers will handle many NMP wastes. Where possible, biotreatment is recommended. Additional information on the biodegradability and aquatic toxicity of NMP is given on Page 16. Incineration or landfilling in a licensed facility are further options when an absorbent has been used to contain an NMP spill.

Health and Physical Hazards

Toxicity and First Aid Measures

Acute Overexposure Effects

Contact with the liquid results in moderate eye irritation and may cause temporary corneal clouding. Skin contact results in mild irritation. Prolonged skin contact may result in redness and dermatitis. Inhalation of the vapors of NMP results in respiratory irritation. Accidental ingestion of the liquid causes gastric disturbances; nausea and vomiting may result.

Chronic Overexposure Effects

In animal studies in rats and mice, NMP was embryotoxic by the oral and intraperitoneal routes at very high dose levels which were close to the LD50. In the dermal exposure study with rats, NMP was only embryotoxic at the high dose level; this effect was attributed to maternal toxicity. Several inhalation studies in rats did not reveal any indication of maternal toxicity or embryotoxicity. In a 2-year inhalation study, NMP did not cause any life-shortening or carcinogenic effects in rats at 0.004 or 0.4mg/l (10 and 100 ppm, respectively). Testicular effects were seen after oral and inhalation exposure at high dose levels in animal studies.

First Aid Measures

Skin: Remove contaminated clothing. Wash affected areas with soap and water. If irritation develops, get medical attention. Launder contaminated clothing before reuse.

Eyes: Wash eyes immediately with running water for 15 minutes. If irritation develops, get medical attention.

Ingestion: If swallowed, dilute with water, and induce vomiting immediately. Never give fluids or induce vomiting if the victim is unconscious or having convulsions. Get immediate medical attention.

Inhalation: Move to fresh air. Aid in breathing, if necessary, and get immediate medical attention.

Exposure Limits

There are no PELs (permissible exposure limits) established by OSHA for NMP. Occasionally, PELs are updated; therefore, refer to your supplier's Material Safety Data Sheet (MSDS) for the most accurate and up-to-date information.

General Personal Protection Data

When handling NMP, the following personal protective equipment should be used: butyl rubber or FEP Teflon® gloves, and safety glasses with side shields. Depending on use, boots, aprons and chemical splash goggles with full face shields may also be required. Following use, gloves should be rinsed with water and discarded.

If vapors or mists are generated, wear a NIOSH/MSHA approved organic vapor/mist respirator.

Safety showers and eyewash fountains should be easily accessible to operating personnel.

Toxicology Test Data

Extensive toxicology data exist for N-Methylpyrrolidone. Refer to your supplier's MSDS for the most up-to-date information.

Transportation, Storage and Handling

Rail Car Unloading

The following information is for top unloading and is provided without guarantee, as the actual conditions of unloading and handling may vary.

BASF Corporation does not recommend unloading N-Methylpyrrolidone through the bottom valve of the rail car for safety and environmental reasons.

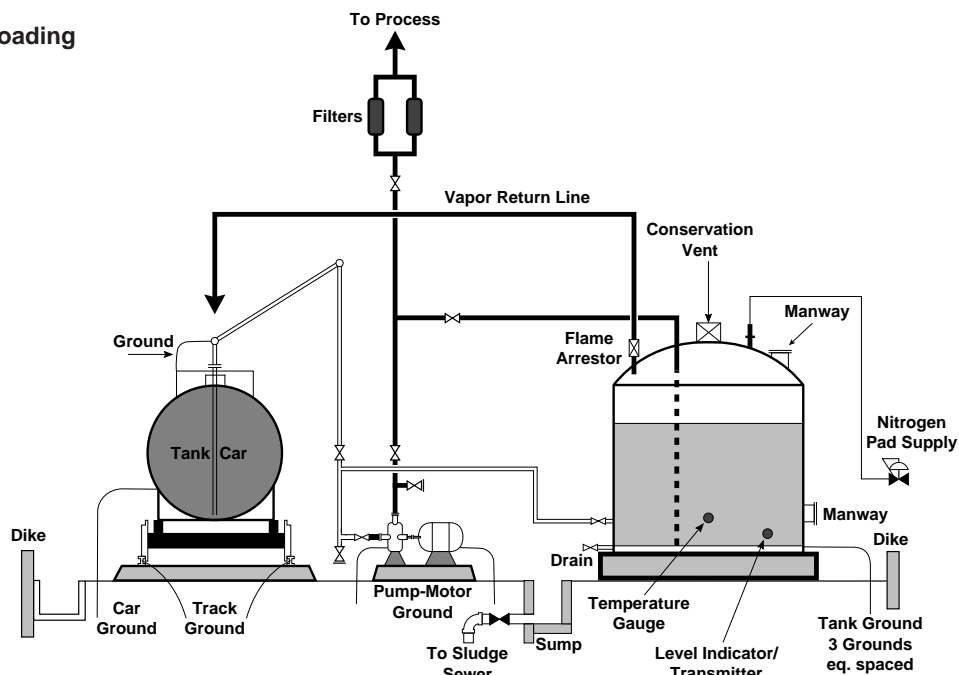
General

Assure that the recommended personal protective equipment is used before proceeding with the unloading operation. See section "Health and Physical Hazards" on Page 5 of this brochure for recommendations.

Unloading procedure

1. Chock the wheels of the rail car. Attach a certified grounding cable to the frame of the car. See Table 1 on Page 10 for recommended ground resistances.
2. Compare the rail car number with the bill of lading to ensure the proper shipment is to be unloaded.
3. Open the standpipe cover and verify that the standpipe valve is in the closed position. Remove the blind-flange and assure that the flange surface is clean.
4. In order to inspect and sample the shipment, remove any pressure by opening the vent valve.
5. Loosen each bolt on the dome cover and loosen the cover, but do not remove the bolts until you are sure the car is vented.
6. Connect the unloading line to the flange of the standpipe using a new gasket. As an alternative to a rigid unloading arm utilizing swivel joints, a metal-braided butyl rubber or Teflon®-lined transfer hose can be used.
7. If a stainless steel flex-hose is used, it is recommended that a ball valve be installed between the standpipe outlet and the flex-hose.
8. If pressure unloading is preferred, connect a nitrogen supply hose to the vent valve of the car. Apply 15 to 20 psig nitrogen to tank car. Once the above pressure is reached, slowly open the standpipe or unloading arm valve.
9. For pump unloading, use centrifugal or positive displacement pumps.
10. Check for leaks at all hose/transfer line connections.
11. Open all valves in the transfer line and the tank inlet valve.
12. If a pump is used for unloading, follow the same procedure until the pump is primed and pumping. Then reduce nitrogen pressure to 1 to 3 psig.
13. Once the rail car is empty, close all valves on the product discharge line, tank car and storage tank.
14. Secure the nitrogen source. Disconnect at the nitrogen inlet and relieve the pressure on the tank car.
15. Before releasing the empty rail car, close the dome cover assuring that the cover gasket is in place and all bolts are tightened. Verify that the vent valve and standpipe are in the closed position.
16. Supervise the entire unloading operation with responsible operating personnel.

Figure 1: Rail Car Unloading



Transportation, Storage and Handling

Tank Truck Unloading

The following information is for unloading N-Methylpyrrolidone and is provided without guarantee, as the actual conditions of unloading and handling may vary.

General

Assure that the recommended personal protective equipment is used before proceeding with the unloading operation. See section "Health and Physical Hazards" on Page 5 of this brochure for recommendations.

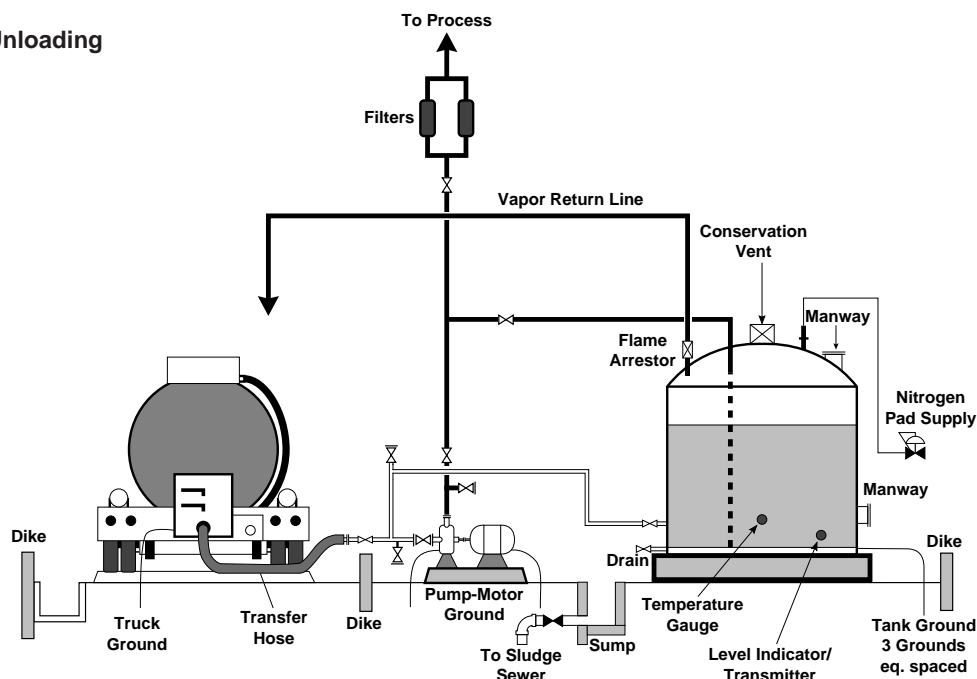
Hose requirements

For the transfer of N-Methylpyrrolidone, it is recommended that a stainless steel flex-hose of a minimum inner diameter of two inches or a steel-braided butyl rubber or Teflon®-lined hose be used. It is recommended that product transfer hoses equipped with "dry disconnect couplings," instead of conventional quick disconnects, be used. This type of coupling reduces product losses or spillage significantly, should accidental disconnection of coupler and adapter occur, or when disconnecting the hose after product transfer.

Unloading procedure

1. Chock the wheels of the tank truck. Attach a certified grounding cable to the frame of the truck. See Table 1 on Page 10 for recommended ground resistances.
2. Compare the trailer number with the bill of lading to ensure that the proper shipment is to be unloaded.
3. If required, record the current product temperature as shown on the built-in dial thermometer.
4. Open the manway. Lack of proper ventilation can result in damage to the tank truck.
5. Assure that the internal and external valves are in the closed position. Place a spill pan underneath the outlet nozzle. Carefully remove the cap from the product outlet nozzle of the compartment, and inspect for cleanliness.
6. Remove the caps from the unloading hose. Connect the unloading hose to the tank truck outlet. Prepare pump for unloading.
7. Open the internal valve and then the external valve and check for leaks.
8. Open all valves in the transfer line and the tank inlet valve.
9. Start the unloading pump. Monitor the liquid level in the tank truck. Check for possible leaks at the transfer hose connections.
10. The entire unloading operation should be supervised continuously by the responsible operating personnel.
11. Once the unloading is completed, close the internal valve first and then the external valve.
12. Secure the transfer pump and close all block valves in the transfer line.
13. Disconnect the unloading hose and place the cap over the product outlet. Cap the open ends of the transfer hose.
14. Close the dome and tighten all bolts. Clean the dome and discharge area. Dispose of all product collected from cleaning or from the spill pan. Do not discharge into waterways or sewage systems.

Figure 2: Tank Truck Unloading



Transportation, Storage and Handling

Drumming Procedure

Product Certification

The tank or tank truck to be drummed must be certified. If a truck shipment is used for drumming, the written certificate of analysis that accompanies the shipment should be used for the drummed material. The lot number shown on the certificate of analysis should be used for the drummed lot as well, unless otherwise specified. If a storage tank is used for the drumming, the tank should be sampled and analyzed in order to establish a certificate of analysis. This certificate is used for the entire drummed lot and a lot number is assigned as specified by the drumming batch sheet.

General

Assure that the recommended protective equipment is used before proceeding with the drumming operation. See section "Health and Physical Hazards" on Page 5 of this brochure for recommendations.

Drum Specifications

It is recommended that 55 gal. steel drums, DOT 17E with Gasket Testing Specification Code 404022 be used.

Drumming Station Requirements:

1. Location
The drumming station should be located in a well-ventilated area indoors with ample access provided for drum and pallet movement by forklifts.
2. Recommended Equipment
A drumming station should consist of the following major components:
 - Gravity conveyor for empty drums
 - Bung alignment machine
 - Drum filling machine
 - Palletizing machine
 - Control instrumentation

3. Drumming Accuracy/Efficiency

The filling system should be designed to fill a 55 gal. drum to within 0.2 lbs. of the required net weight. An automatic tare system should compensate for the empty drum weight during the filling cycle. An automatic dribble feature to reduce the flow rate at the end of the fill cycle will help prevent overcharges. An automatic bung alignment system will help to improve the efficiency of the drumming operation and reduce the possibility of product spills.

4. Drumming Documentation

BATCH SHEET

A drumming batch sheet should be used during the drumming operation. This sheet should contain the following information:

- Product Name
- Lot Number
- Number of drums to be filled
- Target Net Weight
- Sample schedule during the drumming operation

The batch sheet should provide tank inventory records and shift drumming records also.

DRUMMING CHECKLIST

A drumming checklist should be used as a guideline to help eliminate potential problems. All sections of the list should be addressed during the drumming operation. The checklist should be signed and returned to supervision along with the drumming batch sheet.

5. Drum Labels

The product label should be attached to the side of the drum directly below the 3/4-inch bung.

The certificate of analysis label (if required) should be attached to the left of the product label. The labels should be attached to the drum before filling with product.

Drumming Procedure

Operating personnel assigned to the drumming operation should be thoroughly familiar with the physical and toxicological properties and with the recommended "Safe Handling Procedures" outlined in the section "Health and Physical Hazards" of this brochure.

1. Initial Preparations

Assure that an adequate supply of drums, pallets, labels, and bung seals as well as the required personal protective equipment are available.

- Inspect drums for physical damage and assure that the bungs are closed.
- Tighten the 3/4-inch bung to the specified torque as specified on the batch sheet and install a shipping seal.
- Attach the product label and the certificate of analysis label (if required).
(Note: The initial slop drums need only a product label.)
- Line up the drumming line from the appropriate tank or truck.
- Allow the first few drums to enter the staging area and remove the 2-inch bung.
- Inspect each drum internally for cleanliness using a flashlight.
- Set up the drumming instrumentation to the required net weight as per batch sheet.
- Before filling the first drum for customer shipment, flush the entire line through the drumming nozzle. The required amount of flush depends largely on the local piping system and has to be determined by analytical methods.

Transportation, Storage and Handling

2. Drumming Operation

Once the analysis for contaminants shows the required purity, customer drumming can commence. It is essential that only the laboratory give the approval for customer drumming.

- Number each drum sequentially (starting with #1) on the top near the 3/4-inch bung.
- Proceed with filling operation.
- If automated drumming equipment is utilized, follow the manufacturer's operating instructions.
- Draw drum samples as required by the sampling schedule.
- Immediately after a drum is filled and sampled, install the 2-inch bung, and tighten to the required torque as specified in the batch sheet.

Note: In order to apply the correct torque to the 3/4-inch and 2-inch bungs, it is essential to use a torque wrench. Improperly torqued bungs tend to leak.

- Check the top of the drum for any traces of product, and wipe clean using a cloth. It is recommended that portable flammable waste containers with tightly fitting, self-closing lids be used for the temporary waste storage. Final disposal of the contaminated cloth should be done in accordance with state, local and federal requirements.

Scale Weight Verification

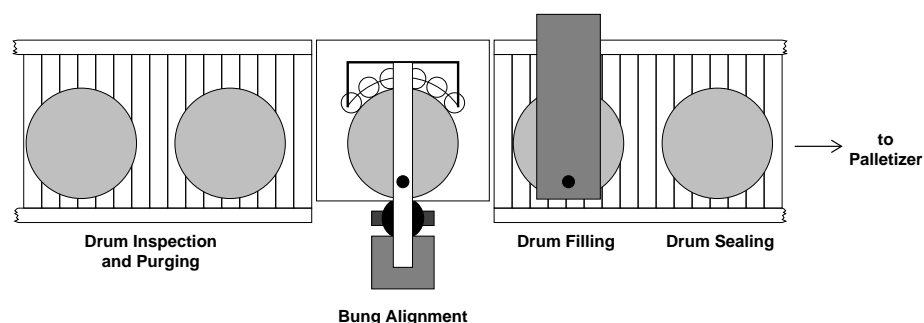
In order to confirm the accuracy of the drum filling weight, the scale accuracy should be verified on a bi-monthly basis and, if necessary, re-calibrated using certified calibration weight standards.

Technical Service

BASF Corporation believes that product responsibility includes both quality control of its products and technical service regarding safe handling and proper use of these products.

We are prepared to assist customers with problems pertaining to the storage and handling of these products.

Figure 3: Drumming Schematic



Equipment

Tanks

NMP can be handled in carbon steel, stainless steel or nickel equipment. Aluminum is suitable for NMP service at ambient temperatures only. Storage at ambient temperatures will not affect NMP. Cool storage conditions and light-colored paint on tanks decrease evaporative losses.

Either vertical or horizontal tanks are suitable for bulk storage and may be located indoors, outdoors or underground. Location of the tank should be in a protected area and in compliance with governmental regulations and engineering practice regarding storage of bulk liquids. The tank should be sized 1.5 times larger than the maximum normal amount received.

Tanks should be equipped with top and side manways. Provisions should be made for a 2-inch diameter or larger flanged bottom outlet, as well as top connections for fill pipe, level gauge, emergency vent and a conservation vent. Dry nitrogen blanketing of storage tanks is the preferred method of moisture control. The dry nitrogen blanket is maintained at a slightly positive pressure by regulating valves so that all air is excluded. This will minimize rusting of the tank interior, which could cause discoloration of the NMP. Typical unloading and storage arrangements are shown in Figures 1 and 2.

Tank foundations should be in accordance with accepted engineering practices and codes. Most states have diking regulations. Be sure to check into state and local regulations governing the diking of areas surrounding NMP storage tanks.

Piping

Storage tanks should be equipped with a fill pipe that extends to within 4 inches of the bottom to prevent static electricity charges from forming. The dip pipe should be provided with a gas bleed hole near the top of the dip pipe to act as a siphon break.

ASTM A53 seamless pipe with gasketed flanges is preferred. Polytetrafluoroethylene (PTFE) machined or envelope-type gaskets are suitable up to 500° F (260° C) depending on manufacturer's recommendation. Flexitallic® type gaskets should be used at higher temperatures. However, schedule 80 pipe with 3,000 lb. screwed forged fittings, assembled without pipe dope and back welded, is acceptable also. All non-back welded, screwed connections should be sealed with a PTFE thread sealant.

Flanged 150 psi rated carbon steel gate valves are recommended. Carbon steel ball valves may also be used. Avoid brass or bronze valves. A wedge ring of PTFE or Kalrez® is preferred for valve stem packing.

Hoses

Stainless steel metal-braided hoses have proved satisfactory for handling NMP. For special applications, PTFE or polyethylene metal-braided hoses may also be used.

Pump

Centrifugal or positive displacement pumps are satisfactory for use with NMP. For clean solvent service, canned motor pumps have been used, providing a leak-free environment from the pump.

Pressure and Vacuum Relief

Storage tanks should have a pressure-vacuum relief vent to prevent the buildup of pressure as NMP is charged into and withdrawn from the tanks. A conservation-type safety vent is advisable. A properly-sized emergency venting device is essential to prevent injury to personnel or damage to the tank if a fire occurs.

Testing

The bulk storage system, tanks and piping should be hydrostatically tested for leaks before introducing NMP. Upon completion of the test, displace the water with nitrogen and dry thoroughly prior to charging with NMP.

Ground Requirements

Each part of the NMP bulk handling system—tanks, pumps, hoses, platforms, tank cars and tank trucks—should be electrically grounded to a ground rod or other approved grounding system. This helps avoid static electricity buildup during solvent pumping operations.

Wire jumpers should be installed at swing joints and other locations to provide an unbroken low-resistance ground circuit.

Recommended ground resistances are shown in the following table:

Table 1:

Service	Maximum Allowable Resistance, Ohms	Grounding
Motors—Hazardous areas	3	Grounding wire in conduit with circuit connectors
Tanks and vessels	2–5	Cable to establish ground
Pipelines with flammables	2–5	Driven rods, cables
Tank cars and rails	2–5	Driven rods, cables

Adequate grounding is required for drum-loading stations, including both the drums and conveyor. The minimum size for grounding cable should be AWG No. 4

Cleaning Storage Tanks

If it is necessary to clean or repair an NMP storage tank, all traces of the solvent, both liquid and vapor, should be removed from the whole system including the pumps, piping and associated equipment. First, the solvent is drained. Then, the entire system is rinsed thoroughly by filling and draining several times with water. After the last water is drained, the entire system is purged with air.

Before performing maintenance that is likely to produce sparks (welding, wire brushing, etc.) the atmosphere in the tank and its immediate vicinity should be checked with an explosimeter to ensure there are no explosive mixtures present.

Cleaning the tank from the outside is preferred. But, if it is necessary to enter the tank, the following additional precautions should be taken, and OSHA Code 29CFR1910.146 should be observed.

1. All piping to the tank is to be disconnected or capped.
2. A positive flow of fresh air is to be provided to the tank.
3. Tank oxygen content is to be tested prior to entry and monitored while personnel are inside the tank.
4. Tank atmosphere is to be rechecked with an explosimeter for explosive mixtures.
5. Personnel entering the tank must have a safety rope and wrist straps attached. The other end of the rope must be tended constantly by personnel outside the tank who will keep personnel inside the tank under observation at all times. Adequate additional personnel should be nearby if assistance is required to withdraw individuals from the tank.

The interior of the tank can be cleaned by brushing down the side walls from top to bottom and removing all debris. As an aid to cleaning, either trisodium phosphate or sodium carbonate can be used in concentrations of 0.5 lbs. per gallon of water. Chemical goggles and gloves should be worn during this procedure. The cleaning solution can be flushed out with water.

Before bringing the system back on stream, it should be tested hydrostatically for leaks. Upon completion of the test, displace the water with nitrogen and dry thoroughly prior to charging with NMP.

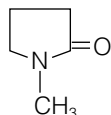
Physical Properties and Compatibility

Table 2: N-Methylpyrrolidone Physical Property Data

Formula:



Structure:



	CGS	ENGLISH	SI
Molecular weight	99.1g/mole	99.1	99.1
Density-Liquid (20°C)	1.03gm/cc	64.3lb/ft ³	1,030kg/m ³
(40°C)	1.02gm/cc	63.6lb/ft ³	1,020kg/m ³
(60°C)	0.99gm/cc	61.8lb/ft ³	990kg/m ³
Boiling point (760 mm Hg)	204.3°C	399°F	477K
Freezing point (760 mm Hg)	-23.6°C	-10.3°F	249.6K
Viscosity (20°C)	1.7cp	4.11lb/ft-hr	1.7mPa-s
(50°C)	1.0cp	2.41lb/ft-hr	1.0mPa-s
(80°C)	0.9cp	2.17lb/ft-hr	0.9mPa-s
Heat of Vaporization (100°C)	122cal/g	219Btu/lb	510kJ/kg
Specific Heat-Liquid (0°C)	0.401cal/g-°C	0.401Btu/lb-°F	1.68kJ/kg-K
(50°C)	0.465cal/g-°C	0.465Btu/lb-°F	1.89kJ/kg-K
(100°C)	0.502cal/g-°C	0.502Btu/lb-°F	2.10kJ/kg-K
Specific Heat-Vapor (25°C)	0.301cal/g-°C	0.301Btu/lb-°F	1.26kJ/kg-K
Vapor Pressure (40°C)	1.0Torr	0.02psi	133Pa
(60°C)	3.5Torr	0.07psi	465Pa
(80°C)	9.5Torr	0.19psi	1270Pa
Refractive Index	1.4700	1.4700	1.4700
Heat of Combustion	7.29kcal/g	13,100Btu/lb	30,500kJ/kg
Flash Point (ASTM-D 93-72)	91°C	196°F	364K
Ignition Temperature (DIN 51794)	245°C	473°F	518K
Flammable limits in air			
Upper	9.5 vol%	9.5 vol%	9.5 vol%
Lower	1.3 vol%	1.3 vol%	1.3 vol%
Thermal conductivity (25°C)	1.63 W/cm-°C	1.13Btu-in/ft ² hr°F	0.163 W/m-K
Hansen Solubility parameters:			
δd	8.8 (cal/cm ³) ^{1/2}		18.0 (J/cm ³) ^{1/2}
δp	6.0 (cal/cm ³) ^{1/2}		12.3 (J/cm ³) ^{1/2}
δh	3.5 (cal/cm ³) ^{1/2}		7.2 (J/cm ³) ^{1/2}
δo	11.2 (cal/cm ³) ^{1/2}		22.9 (J/cm ³) ^{1/2}
Kauri-Butanol value (ASTM D1133-83)	>300		

Physical Properties and Compatibility

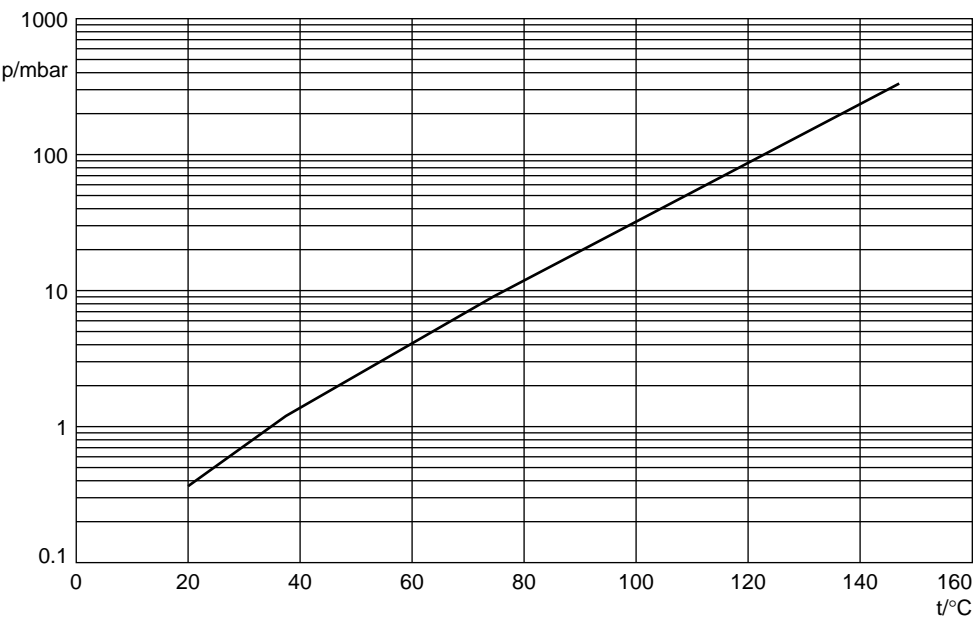


Figure 4: Vapor Pressure

Antoine Constants for $\ln p = A - \frac{B}{(C + t)}$

p in bar
t in °C
A = 10.2671
B = 4290.30
C = 214.11

The vapor pressure of NMP at 20°C is 0.316 mbar. This means that in a dry atmosphere (at a temperature of 20°C and a pressure of 1013 mbar) a maximum NMP concentration of 1.29 mg/l respectively 312 ppm (vol) can be achieved.

Table 3: Flash Points of Mixtures of NMP and Water

The following values were determined according to ASTM-D 93-72

Concentration (wt. %)		Flash Point
NMP	H ₂ O	t/°C
100	0	91
98	2	93
95	5	97
90	10	104
80	20	118

Physical Properties and Compatibility

NMP is a widely used industrial solvent for natural and synthetic plastics, waxes, resins and various types of paints. It dissolves polymers, such as cellulose derivatives, polyamides, polyimides, polyesters, polystyrene, polyacrylonitrile, polyvinyl chloride, polyvinyl pyrrolidone, polyvinyl acetate, polyurethanes, polycarbonates, polyethersulfones, polysulfones, polyethers and many copolymers (see Table 4).

Table 4: Solubility of Plastics in NMP at Ambient Temperatures

Polyethylene	—
Polypropylene	—
Polystyrene	+
Styrene-acrylonitrile-copolymer	+
Acrylonitrile-butadiene-styrene-polymer	+
Polymethyl methacrylate	+
Polyvinyl chloride	+
Polyamide	— (+ at 200°C)
Polyurethane	○
Polycarbonates	○ (+ at 80°C)
Polychloroprene	+
Polytetrafluoroethylene	—
Polyvinyl pyrrolidone	+
Polysulfone	+
Polyethersulfone	+
Polyphenylene sulfide	—
Polyacrylonitrile	+
Polyesters	+
Epoxy resins	○
Polyethylene glycol	+

+ dissolved (>10%)

○ sparingly soluble/swollen

— undissolved/resistant

Since no two generic types of plastics are exactly alike, it is not prudent to draw direct conclusions from the above-described behavior. Therefore, data from Table 4 should be used only as a starting point for selecting materials to be tested in specific application trials.

NMP is miscible with the corresponding monomers and with conventional organic solvents and water. As a result, the best possible solvent blend for a given application can be found.

Table 5: Determination of Resin Solubility in NMP (according to DIN 55955)

Cellulose derivatives	●
Cellulose nitrate	●
Cellulose acetate	●
Cellulose acetobutyrate	●
Ethyl cellulose	●
Chlorinated rubber	●
Vinyl chloride rubber	●
Vinyl chloride copolymers	●
Polyvinyl chloride	●
Laroflex MP®	●
Lutofan 200L®	●
Vinyl chloride vinylacetate copolymers	●
Vinyl chloride maleate copolymers	●
Polyvinyl acetate	●
Polyvinyl ethers	●
Polyacrylonitrile	●
Polyacrylates	●
Acronal 4 F	●
Acronal 700L®	●
Lumitol®	●
Luprenal®	●
Polymethyl methacrylate	●
Styrene polymers	●
Suprapal®	●
Emu-powder®	●
Styrene acrylic acid copolymers	●
Vinyltoluene acrylic acid copolymers	●
Polyisobutylene	●
Oppanol B3®	○
Lutonal IC®	◐
Styrene maleate resins	●
Suprapal AP®	●
Suprapal TP®	●
Unmodified alkyd (phthalate) resins	●
Phtalopal PP®	●
Phtalopal NP®	●
Cyclohexanone resins	●
Laropal K80®	●
Urea resins	●
Plastopal®	●
Melamine resins	●
Luwipal®	●
Phenolic resins	●
Thermosetting acrylic resins	●
Larodur®	●
Epoxy resins	●
Modified alkyd resins	●
Natural resins	●
Rosin	●
Ester gum	●
Manila copal	●
Shellac	●
Drying oils	●
Linseed oil	●
Tung oil	●
Linseed oil stand oil	○
Tung oil stand oil	○
Plasticizers	●
Castor oil	●
Butyl stearate	●
Tritolyl phosphate	●
Palatinol®	●
Plastomoll®	●
Palamoll®	●
Plastigen G®	●
Chlorinated paraffins	●

● soluble

○ insoluble

◐ sparingly soluble or swellable

Physical Properties and Compatibility

Materials of Construction

Several rigid plastics, including UHMW polyethylene, polytetrafluoroethylene, nylon 6/6 and acetal copolymer, were tested by partial immersion in NMP at 120°F (49°C) for six weeks. All samples showed less than one percent weight gain and dimensional stability within one percent.

Several elastomers were also tested by partial immersion in NMP at 120°F (49°C) for six weeks. Silicone rubber and butyl rubber showed the best weight and dimensional stability.

Although not tested in our laboratories, Kalrez® has provided good field results when in service with NMP.

Whether the above materials will prove satisfactory for any given application depends on many factors, such as the temperatures, pressure and purity of the NMP being handled, as well as the processing and application of the materials. Therefore, the above report of test results should be used only as a starting point for selecting materials to be tested in specific application trials.

Biodegradability and Aquatic Toxicity

Introduction

Biodegradation refers to the biologically (usually bacteria) catalyzed breakdown of organic chemicals. When dissolved oxygen is present, as it typically is in a biologically healthy stream or river, the end products are carbon dioxide (CO₂) and water (H₂O). In practice, complete oxidation is rare. More commonly, a partial breakdown occurs resulting in the formation of CO₂, H₂O and metabolites.

Most chemicals will cause environmental damage if released untreated. The damage can take many forms depending on the chemical involved. Direct toxic effects may take the form of:

- poisoning of aquatic species through short single dose exposure (acute toxicity).
- poisoning of aquatic species due to persistent non-degraded toxicants (chronic toxicity).
- poisoning of higher members of the food chain through bioaccumulation in aquatic species.

Indirect toxic effects may take the form of:

- oxygen demand overloading, causing dissolved oxygen levels in the water to drop below the level required by local fish species.
- high algae growth rates resulting in lake eutrophication.

Prevention measures require minimizing both the amount and toxicity of the wastewater discharges.

The need for treatment of chemicals prior to their release is obvious. In turn, this makes the need for a means of predicting the degree of treatment obvious. However, considering the wide range of circumstances that may be present, the form of the measurements needed to make these predictions is far from obvious. Our measurement, biodegradability, is not an absolute intrinsic property. Its value, and the method involved in determining that value, are dependent on the particular conditions which have necessitated the treatment.

For example, a chemical which might be released inadvertently to a receiving water (a spill) would have different properties of interest than a chemical intentionally released to a wastewater treatment plant (aquatic toxicity vs. BOD). Similarly, the levels of treatment required and the ultimate efficiency of treatment achieved would differ as would the tests required to measure them.

Measures of Biodegradability

Biodegradability is a function of the type and amount of bacteria available, the structure of the chemical being treated, various environmental factors (oxygen, pH, etc.), and the contact time. Different treatment situations will involve different combinations of the above. Thus, it is understandable that many different types of tests exist.

Primary biodegradability was an early test method established to measure the loss of a particular property of interest (surface tension or foaming). This test used high concentrations of acclimated bacteria (adapted to the chemical). Contact times are on the order of one day. Since only a small structural change is required in the test chemical to cause loss of a physical property, this type of test generally gives very extensive biodegradability even though most of the molecular makeup of that chemical remains untouched. For this reason, this type of test has fallen out of use.

The familiar BOD (Biological Oxygen Demand) test is an example of a respirometric (oxygen consumption) measurement of biodegradability. It is the most commonly used test to describe wastewater treatment plant performance. By measuring oxygen consumption, these tests provide a better indication of the extent of degradation of the chemical structure than does loss of a property of the chemical. These tests are often reported as percentages of theoretically complete oxidation (ThOD). Accordingly, the biodegradability values are generally lower even though the extent

of treatment may often be greater. This type of test varies in the conditions under which it is run; however, it is generally run with low concentrations of bacteria which may or may not be acclimated and contact times on the order of 1–5 days.

A third general type of test takes the respirometric test a step further by increasing the contact time to 30 days or longer (virtually ensuring that the bacteria are acclimated). The objective of this test is to measure the ultimate biodegradability (greatest possible extent of degradation). This testing simulates the environmental fate of chemicals in a receiving water. The biodegradability values are usually higher than the shorter term BOD type tests.

Biodegradability can also be measured by monitoring other properties or descriptions of the test chemical. Chemical Oxygen Demand (COD) measures (for practical purposes) the complete oxidation of a chemical to CO₂ and H₂O. Thus biodegradability measured by COD is generally lower than by any other test. The ratio of the BOD to COD gives the percentage of the molecule that can be biologically oxidized. A value of 0.3 or greater is considered good.

Total Organic Carbon (TOC) is sometimes used to describe a molecule. Values of biodegradability obtained by monitoring that parameter generally lie between BOD and COD values.

Obviously, it is essential that the test conditions be specified when citing biodegradability numbers. When comparing values from different sources, it is vital to ensure that the values are describing the same test.

Biodegradability and Aquatic Toxicity

Measures Of Aquatic Toxicity

The harmful effect of a chemical released into a receiving water is dependent on its concentration in that water. All chemicals are toxic if their concentration is high enough. Therefore, it is necessary to determine a "safe" concentration. The tests for doing this generally use various forms of aquatic life as indicators of toxicity. The values reported usually show the chemical concentration at which 50% of the test organisms would be expected to die. The common term for this is the LD50. Because the life forms used in this testing vary greatly in their sensitivity to chemicals, the test results also vary greatly. Again, it is important to specify the conditions used in the test and to make sure that conditions are identical when comparing values. In general, LD50 values <10ppm are considered toxic. Values between 10 and 100 ppm are somewhat toxic. And values >100 ppm are considered non-toxic.

NMP Biodegradability

River die-away tests were conducted by Chow & Ng (1). These tests simulate the fate of a compound in a receiving water. Using an initial concentration of 100 ppm NMP, they found 95% removal as measured by specific compound analysis and 45% removal as measured by COD. These determinations were conducted after two weeks exposure. The low COD removal compared to the specific compound removal indicates a high degree of molecular modification (probably nitrogen-carbonyl bond splitting) and a lesser amount of mineralization (conversion to CO₂ and H₂O). Thus, while the compound was no longer present as NMP, the compound was still present as modified fragments.

An additional test by the same people using a semi-continuous activated sludge system acclimated for five days showed seven-day biodegradability of 95% by specific analysis.

In similar tests, Matsui (2) used a semi-continuous system to measure TOC, COD

and specific compound (by GC) removals. These results all showed >92% removal (regardless of the parameter being measured) within 24 hours starting with concentrations ranging from 92 ppm to 210 ppm NMP.

Rowe and Tullos (3) studied biodegradability of NMP in acclimated and unacclimated, static and continuous flow, laboratory activated sludge systems. The tests were run at 300 and 1000 ppm of NMP; the continuous cells had an 18-hour hydraulic residence time. The results indicate that NMP was largely degraded (more than 98% destruction with 90% TOC reduction in the continuous cells), and that significant acclimation is easily achieved (more than 75% TOC reduction in 24 hours in unacclimated static tests).

Respirometric tests provided by BASF AG show a BOD₅ (acclimated) of 1.2 wt/wt. Compared to the Theoretical Oxygen Demand (ThOD = COD) of 2.18, this gives a BOD/COD ratio of 0.55, which is quite biodegradable.

As an indication of the impact of NMP on an activated sludge treatment system, the *Handbook of Environmental Data* (4) lists a no-effect level (LD0) for NMP using the bacterium *Pseudomonas* as 5g/l. This demonstrates that NMP is non-toxic to treatment systems even at fairly high concentrations. BASF AG tests indicate that activated sludge wastewater treatment systems will continue to function efficiently at NMP concentrations as high as 2000 ppm.

BOD tests conducted by BASF AG show that when using unacclimated bacteria, NMP is biodegradable; BOD₅ = 0.76 wt/wt. However, acclimation significantly increased the biodegradability; BOD₅ = 1.15 wt/wt. Long-term BOD tests show substantial biodegradability BOD₂₀ = 1.2 wt/wt.

All of the above tests show that in acclimated systems or systems that could acclimate during the normal contact time (as in the case of a spill to a receiving water), NMP is readily biodegradable. There are indications

that in unacclimated, short contact time systems (single dose exposure in municipal treatment plants) virtually complete elimination of NMP can be expected.

Aquatic Toxicity

GAF data for various species of fish list the LC50 as ranging from 832-3048 ppm. Even the lowest value is well within the non-toxic range.

Conclusions

NMP is nearly non-toxic to most aquatic life and can be readily degraded by typical wastewater treatment plant organisms. It is, accordingly, an advantageous solvent to use whenever the possibility of discharge into an aqueous stream exists.

References

1. "The Biodegradation of N-Methyl-2-Pyrrolidone in Water by Sewage Bacteria," S.T. Chow and T.L. Ng *Water Research*, Vol. 17, pp. 117-118 (1983).
2. "Activated Sludge Degradability of Organic Substances in the Waste Water of the Dashima Petroleum and Petrochemical Industrial Complex in Japan," S. Matsui, T. Murakimi, T. Sasaki, Y. Hirose, and Y. Iguma, *Progress in Water Technology*, Vol. 7, Nos. 3/4, pp. 645-659 (1975).
3. "Lube Solvents No Threat to Waste Treatment," E.H. Rowe and L.F. Tullos, Jr., *Hydrocarbon Processing*, 59, pp. 93-65 (October 1980)
4. *Handbook of Environmental Data on Organic Chemicals*. 2nd Ed., Karel Verschueren, Van Nostrand, Reinhold Co., N.Y. 1983, p. 873.

Analytical Procedures

Procedures

Assay: This method is used to determine the assay of NMP by means of gas chromatographic analysis.

The following labware and reagents are required for this method of determination:

Gas chromatograph (GC) equipped with a FID detector
 Capillary column cross linked methyl silicone
 0.33 micron film thickness
 25 meters length
 0.20 mm diameter

One 10 microliter syringe

The procedure for the determination of the assay of NMP is as follows:

GC conditions:

Initial temperature	100°C
Final temperature	180°C
Initial hold	2 minutes
Final hold	2 minutes
Rate	10°C/minute
Carrier gas flow	0.5 cc/minute

Allow the GC to reach equilibrium.

Inject 0.1 microliters of sample.

Components:

	<u>Relative retention time</u>
gamma butyrolactone	0.68
NMP	1.00
1,3-dimethylpyrrolidone	1.03
1,4-dimethylpyrrolidone	1.08

Free Amines (Method I): This method is used to determine the amine content of aqueous solutions by means of titration with 0.01 N hydrochloric acid.

The following labware and reagents are required for this method of determination:

0.01 N hydrochloric acid (HCl)
 Erlenmeyer flask
 Methyl red indicator
 Burette (50 mL)

The Method I procedure for the determination of the methylamine content is as follows:

Weigh 40 grams of sample into 40 mL demineralized water.

Add several drops of methyl red indicator.

Titrate with 0.01 N HCl until color changes from yellow to red.

The methylamine content is calculated from the following:

$$\text{amine, ppm} = \frac{\text{mL of HCl} \times 3.106 \times 100}{\text{Weight of sample (g)}}$$

Note: 3.106 is the amine factor for monomethylamine.

Free Amines (Method II): This method is used to determine the amine content of aqueous solutions by means of ion chromatography.

The following labware and reagents are required for this method of determination:

Eluent—0.025 N hydrochloric acid (HCl)
 Regenerant—0.1 M tetramethylammonium hydroxide pentahydrate
 Ion chromatograph
 Cation suppressor column
 Cation separator column—Dionex CS 12
 Monomethylamine (MMA)
 Dimethylamine (DMA)
 Trimethylamine (TMA)

The Method II procedure for the determination of the methylamine content is as follows:

Ion chromatograph conditions:

Eluent flow rate	1 mL/minute
Injection loop	100 microliters
Regen Flow	3 to 5 mL/minute

Prepare standards:

MMA	2 ppm
DMA	4 ppm
TMA	10 ppm

Inject 100 microliters of each standard.

Prepare Sample: Add 20 mL of sample to a 100 mL volumetric flask and dilute to the mark with nano-pure water.

Inject 100 microliters of sample.

The amount of methylamine is calculated from the following:

$$\text{amine, ppm} = \frac{(\text{Pk Hgt. of amine}) \times (\text{Conc. of the Standard})}{\text{Pk Hgt. of standard}} \times \text{dilution factor}$$

APHA Color: This method is used to determine the APHA color of NMP.

The following labware and reagents are required for this method of determination:

Platinum-cobalt stock solution (APHA Number 500)

Nessler tubes (100 cm)

The procedure for the determination of APHA color is as follows:

Prepare an APHA 50 color solution by diluting the platinum-cobalt stock solution 10 to 1.

Fill one Nessler tube with sample and another with the APHA 50 standard. View vertically in a light box and compare colors.

Analytical Procedures

Water: This method is used to determine the water content of NMP by means of Karl Fischer titration.

The following labware and reagents are required for this method of determination:

Karl Fischer titrator

Methanol

Karl Fischer (KF) reagent

The procedure for the determination of water content is as follows:

Add methanol to the Karl Fischer titration flask.

Titrate with KF reagent to an electrometric endpoint.

Add 10 grams of sample to the titration flask. Titrate with KF reagent to an electrometric endpoint.

The amount of water is calculated from the following:

$$\text{Water, \%} = \frac{\text{mL KF reagent} \times \text{KF factor} \times 100}{\text{Weight of sample}}$$

Anions: This method is used to determine the chloride, nitrate, phosphate and sulfate content of aqueous solutions by means of ion chromatography.

The following labware and reagents are required for this method of determination:

Eluent-1.8 mM sodium carbonate/1.7 mM sodium hydrogen carbonate

Regenerant-17.8 megaohm water or better
Ion chromatograph

Anion suppressor column

Anion separator column-Dionex AS 3

The procedure for the determination of the anion content is as follows:

Ion chromatograph conditions:

Eluent flow rate	1 mL/minute
Injection loop	100 microliters
Regen Flow	9 mL/minute

Prepare 50 ppb anion standard.

Inject 100 microliters of each standard.

Prepare sample: Add 20 mL of sample to a 100 mL volumetric flask and dilute to the mark with nano-pure water.

Inject 100 microliters of sample.

The amount of anion is calculated from the following:

$$\text{anion, ppm} = \frac{(\text{Pk Hgt. of anion}) - (\text{Pk Hgt. of Blk})}{(\text{Pk Hgt. of standard}) - (\text{Pk Hgt. of Blk})} \times \frac{\text{dilution factor}}{\text{Conc. of the Standard}}$$

Trace Metals: This method is used to determine the following trace metals content of NMP by means of ICP/MS: aluminum, antimony, arsenic, boron, calcium, chromium, copper, gold, iron, lead, magnesium, manganese, nickel, potassium, sodium, tin, titanium and zinc.

The following labware and reagents are required for this method of determination:

ICP/MS

Ultra pure water

Ultra pure nitric acid

Indium internal standard

The procedure for the determination of the trace metal content is as follows:

Standard Preparation: Prepare standards using 1% nitric acid solution and 20 ng/gram indium internal standard.

Sample Preparation: Slowly evaporate one gram of sample in a quartz dish.

Digest remains with 2 mL nitric acid.

Add water to final weight of 10 grams.

Analyze the individual elements using the prepared solutions and blanks by ICP/MS.

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